

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] however, the front face of a ceramic base material on which the corrosion-resistant member currently used conventionally consists of the nature sintered compact of an aluminum oxide -- thermal spraying and CVD -- the [periodic-table] -- the [2a group or] -- when the anticorrosion material which consists of 3a group element compound was made to put and it formed, it was about 95%, and since about 90% and the maximum also had many open pores, the relative density of anticorrosion material had the problem that corrosion resistance was inadequate for them

[0006] Moreover, since the thickness was as thin as about 20-100 micrometers, it exhausted for a short time, and the anticorrosion material put on a ceramic base-material front face by methods, such as thermal spraying and CVD, also had the problem that there was no **** in use of the result long period of time.

[0007] Furthermore, when anticorrosion material is made to put on a ceramic base-material front face by methods, such as thermal spraying and CVD, the adhesion of a ceramic base material and anticorrosion material is low, While it will originate in both coefficient-of-thermal-expansion difference between a ceramic base material and anticorrosion material and thermal stress will occur if heat is impressed to a ceramic base material and anticorrosion material at the time of use since a coefficient-of-thermal-expansion difference is between a ceramic base material and anticorrosion material It also has the problem that anticorrosion material will exfoliate more easily than a ceramic base-material front face, and the function as a corrosion-resistant member will lose with this thermal stress.

[0008] this invention was thought out in view of the above-mentioned trouble, the purpose makes a ceramic base-material front face join firmly thick anticorrosion material with high relative density, and it is in offering the corrosion-resistant member with which prolonged use can be presented.

PRIOR ART

[Description of the Prior Art] Use of plasma, such as a dry etching process, plasma jet flame coating, etc. of semiconductor manufacture, is progressing quickly in recent years. For example, in the semiconductor manufacture process, the fluorine system and the halogen system corrosive gas of ***** are especially used abundantly from the reactant height as a deposition, the object for etching, or an object for cleaning in the plasma process.

[0003] moreover, the front face of the ceramic base material which consists of the former to the nature sintered compact of an aluminum oxide in the portion in contact with the aforementioned gas and plasma in equipment, such as a wall, in order to prevent the corrosion by gas or plasma -- the [periodic-table] -- the [2a group or] -- the corrosion-resistant member to which the anticorrosion material which consists of a 3a group element compound was joined is used

[0004] in addition, the front face of a ceramic base material on which the aforementioned corrosion-resistant member generally consists of the nature sintered compact of an aluminum oxide -- the -- the [2a group or] -- it is formed by making the thickness of 20-100 micrometers put 3a group element compound by thermal spraying or the method of CVD

[0005]

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the corrosion-resistant member suitable for ** implements, such as wall material in the plasma treatment equipment used for it manufacturing a semiconductor device as for which that high corrosion resistance is required from a fluorine system, the corrosive gas of *****, or its plasma, for example, and membrane formation equipment, and supporter material which supports Si substrate.

[0002]

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[0006] Moreover, since the thickness was as thin as about 20-100 micrometers, it exhausted for a short time, and the anticorrosion material put on a ceramic base-material front face by methods, such as thermal spraying and CVD, also had the problem that there was no **** in use of the result long period of time.

[0007] Furthermore, when anticorrosion material is made to put on a ceramic base-material front face by methods, such as thermal spraying and CVD, the adhesion of a ceramic base material and anticorrosion material is low, While it will originate in both coefficient-of-thermal-expansion difference between a ceramic base material and anticorrosion material and thermal stress will occur if heat is impressed to a ceramic base material and anticorrosion material at the time of use since a coefficient-of-thermal-expansion difference is between a ceramic base material and anticorrosion material It also has the problem that anticorrosion material will exfoliate more easily than a ceramic base-material front face, and the function as a corrosion-resistant member will lose with this thermal stress.

[0008] this invention was thought out in view of the above-mentioned trouble, the purpose makes a ceramic base-material front face join firmly thick anticorrosion material with high relative density, and it is in offering the corrosion-resistant member with which prolonged use can be presented.

[0009]

[Means for Solving the Problem] the ceramic base-material front face on which this invention consists of the nature sintered compact of an aluminum oxide -- the [periodic-table] -- the [2a group or] -- the corrosion resistance to which the anticorrosion material which consists of the sintered compact which makes a principal component at least one sort of 3a group element was joined -- it is a member and is characterized by allotting the interlayer who consists of the sintered compact which contains the aforementioned ceramic base-material component and an anticorrosion material component between the aforementioned ceramic base material and anticorrosion material

[0010] Moreover, it is characterized by the coefficient-of-thermal-expansion difference of the aforementioned ceramic base material, an interlayer and the aforementioned anticorrosion material, and an interlayer carrying out in 1 ppm/degree C or less, respectively.

[0011] Furthermore, it has two-layer structure at least, and is characterized by the thing of the 1st interlayer to whom the aforementioned interlayer touches a ceramic base material, and the 2nd interlayer who touches anticorrosion material for which the 1st interlayer of the above is doing the ceramic base-material component, and 55 to 95 volume % and the 2nd interlayer are doing 55-95 volume % content of an anticorrosion material component.

[0012] Furthermore, the aforementioned anticorrosion material is characterized by relative density being 98% or more.

[0013] Furthermore, the aforementioned anticorrosion material is characterized by thickness being 200 micrometers or more again.

[0014] The ceramic base material which consists of the nature sintered compact of an aluminum oxide according to the corrosion-resistant member of this invention, the [periodic-table] -- the [2a group or] -- the anticorrosion material which consists of a sintered compact which makes a principal component at least

one sort of 3a group element It consists of the sintered compact containing the aforementioned ceramic base-material component and the anticorrosion material component, junction nature is good to both ceramic base material and anticorrosion material, and since it joined through the interlayer who has a coefficient of thermal expansion among both, junction of the anticorrosion material to a ceramic base material is very strong. Therefore, though the heat at the time of use is impressed to a corrosion-resistant member, anticorrosion material does not exfoliate from a ceramic base material, and a corrosion-resistant member can be used, always being stabilized.

[0015] the corrosion resistance at the time of impression of heat when the coefficient-of-thermal-expansion difference of a ceramic base material, an interlayer and anticorrosion material, and an interlayer is especially carried out in 1 ppm/degree C or less, respectively -- the junction nature of the ceramic base material of a member and anticorrosion material can be made with a much more good thing, and it becomes possible to use a corrosion-resistant member for stability further

[0016] Moreover, the aforementioned interlayer is made into at least 2 layer structures of the 1st interlayer who touches a ceramic base material, and the 2nd interlayer who touches anticorrosion material. If a ceramic base-material component is done at the 1st interlayer of the above and 55 to 95 volume % and the 2nd interlayer are made to do 55-95 volume % content of an anticorrosion material component It becomes possible considering junction of anticorrosion material [as opposed to / can strengthen the bonding strength between the ceramic base material, between the 1st interlayer and anticorrosion material, between the 2nd interlayer and the 1st, and 2nd interlayer, and / a ceramic base material by this] as a very firm thing to use a corrosion-resistant member for stability further.

[0017] furthermore, the aforementioned anticorrosion material -- the [periodic-table] -- the [2a group or] -- since it is formed with the sintered compact which makes a principal component at least one sort of 3a group element, if relative density of anticorrosion material can be made into 98% or more and relative density of anticorrosion material is made into 98% or more, existence of an open pore will almost be lost and corrosion resistance will become what was extremely excellent

[0018] furthermore, the aforementioned anticorrosion material -- the [periodic-table] -- the [2a group or] -- since it is formed with the sintered compact which makes a principal component at least one sort of 3a group element, if the thickness of anticorrosion material can be formed in a thick thing 200 micrometers or more and thickness of anticorrosion material is set to 200 micrometers or more, it will become possible to present prolonged use from exhaustion of anticorrosion material taking time

[0019]

[Embodiments of the Invention] The gestalt of operation of this invention is explained in detail below.

[0020] drawing 1 -- the corrosion resistance of this invention -- the perspective diagram showing 1 operation gestalt of a member -- it is -- the aforementioned corrosion resistance -- the [ceramic base-material / which a member 1 turns into from the nature sintered compact of an aluminum oxide / 2, and periodic-table] -- the [2a group or] -- it consists of interlayers 4 inserted between the anticorrosion material 3 which consists of the sintered compact which makes a principal component at least one sort of 3a group element, and the aforementioned ceramic base material 2 and the anticorrosion material 3

[0021] the aforementioned ceramic base material 2 -- from the nature sintered compact of an aluminum oxide -- changing -- corrosion resistance -- it acts on a member 1 as a base material for giving intensity and toughness, and prevents that a chip and a crack arise at the time of anchoring to semiconductor fabrication machines and equipment etc., or washing

[0022] if the three-point flexural strength sets to 150 or more MPas and fracture toughness sets the aforementioned ceramic base material 2 to more than 2MPa(s) and room -- corrosion resistance -- the mechanical strength of a member 1 can be made into a strong thing, and it can prevent effectively that a chip, a crack, etc. occur at the time of the installation to semiconductor fabrication machines and equipment etc. Therefore, as for the aforementioned ceramic base material 2, what 150 or more MPas and fracture toughness do for the three-point flexural strength to more than 2MPa(s) and room is desirable.

[0023] moreover -- if the purity of an alumina makes the ceramic base material 2 which consists of the aforementioned nature sintered compact of an aluminum oxide 95 % of the weight or more -- corrosion resistance -- in case it uses for semiconductor fabrication machines and equipment etc. as a member 1, it can prevent effectively an impurity mixing in a semiconductor device and having a bad influence on the property of a semiconductor device Therefore, as for the ceramic base material 2 which consists of the aforementioned nature sintered compact of an aluminum oxide, what the purity of an alumina considers as 95 % of the weight or more is desirable.

[0024] Furthermore, as for the aforementioned ceramic base material 2, the anticorrosion material 3 is

joined to the front face through the interlayer 4.

[0025] the aforementioned anticorrosion material 3 -- the [periodic-table] -- the [2a group or] -- it consists of the sintered compact which makes a principal component at least one sort of 3a group element, and sintered compacts, such as YAG (yttrium aluminum garnet), a yttria, a magnesia, and a spinel, can specifically use it suitably

[0026] The anticorrosion material 3 which consists of sintered compacts, such as Above YAG, a yttria, a magnesia, and a spinel fluorine system gas, such as SF₆, CF₄, CHF₃, ClF₃, NF₃, C₄F₈, and HF, Cl₂, HCl, BCl₃, the chlorine-based gas of CCl₄ grade, or Br₂, HBr, It can prevent effectively that the wallplate of semiconductor fabrication machines and equipment etc. receives corrosion by providing the corrosion resistance which was excellent to the plasma of halogen system corrosive gases, such as bromine system gas of BBr₃ grade, or these halogens system corrosive gas, and using it for semiconductor fabrication machines and equipment etc.

[0027] the aforementioned anticorrosion material 3 -- the [periodic-table] -- the [2a group or] -- since it is formed with the sintered compact which makes a principal component at least one sort of 3a group element, if relative density of the anticorrosion material 3 can be made into 98% or more and relative density of this anticorrosion material 3 is made into 98% or more, existence of an open pore will almost be lost and corrosion resistance will become what was extremely excellent Therefore, what relative density of the aforementioned anticorrosion material 3 is made into 98% or more for is desirable.

[0028] moreover, the aforementioned anticorrosion material 3 -- the [periodic-table] -- the [2a group or] -- since it is formed with the sintered compact which makes a principal component at least one sort of 3a group element, if the thickness of the anticorrosion material 3 can be formed in a thick thing 200 micrometers or more and thickness of the anticorrosion material 3 is set to 200 micrometers or more, it will become possible to prevent prolonged use from exhaustion of the anticorrosion material 3 taking time

[0029] Furthermore, the aforementioned anticorrosion material 3 is joined to the front face of the ceramic base material 2 through the interlayer 4.

[0030] The aforementioned interlayer 4 consists of the sintered compact containing ceramic base-material 2 component and anticorrosion material 3 components, and it becomes possible to join the anticorrosion material 3 to the ceramic base material 2 very firmly to both ceramic base material 2 and anticorrosion material 3, since junction nature is good.

[0031] Moreover, when making it the coefficient of thermal expansion become between the coefficient of thermal expansion of the ceramic base material 2, and the coefficients of thermal expansion of the anticorrosion material 3, there is no big coefficient-of-thermal-expansion difference of four interlayer who consists of the sintered compact containing the ceramic base-material 2 aforementioned component and anticorrosion material 3 components between the ceramic base material 2 and the anticorrosion material 3, and an interlayer 4, therefore, corrosion resistance -- it is possible for big thermal stress not to occur between the ceramic base material 2 and the anticorrosion material 3, and an interlayer 4, though heat is impressed at the time of use of a member 1, and to join the anticorrosion material 3 to the ceramic base material 2 firmly -- becoming -- consequently, corrosion resistance -- a member 1 can be used, always being stabilized

[0032] the corrosion resistance at the time of impression of heat when the coefficient-of-thermal-expansion difference of the aforementioned ceramic base material 2, an interlayer 4 and the anticorrosion material 3, and an interlayer 4 is especially carried out in 1 ppm/degree C or less, respectively -- the junction nature of the ceramic base material 2 of a member 1, and the anticorrosion material 3 -- a much more good thing -- it can make -- corrosion resistance -- it becomes possible to use a member 1 for stability further Therefore, what is done for the coefficient-of-thermal-expansion difference of the aforementioned ceramic base material 2, an interlayer 4 and the anticorrosion material 3, and an interlayer 4 in 1 ppm/degree C or less, respectively is desirable.

[0033] Furthermore, as shown in drawing 2, the thing of 2nd interlayer 4b which touches 1st interlayer 4a which touches the ceramic base material 2, and the anticorrosion material 3 made for the 2nd interlayer 4b to do 55-95 volume % content of an anticorrosion material component is desirable [the aforementioned interlayer 4] while considering as two-layer structure at least and making this 1st interlayer 4a do 55-95 volume % content of a ceramic base-material component.

[0034] The aforementioned interlayer 4 is made into at least 2 layer structures of 2nd interlayer 4b which touches 1st interlayer 4a which touches the ceramic base material 2, and the anticorrosion material 3. To this 1st interlayer 4a, an anticorrosion material component 55-95 volume % content for a ceramic base-material component at 55 to 95 volume %, and 2nd interlayer 4b And if it carries out, since the ceramic

base-material component contains so much in the 1st interlayer 4a, Since 1st interlayer 4a and the ceramic base material 2 are joined firmly and the anticorrosion material component contains so much in the 2nd interlayer 4b, Join firmly 2nd interlayer 4b and the anticorrosion material 3, and since 1st interlayer 4a and 2nd interlayer 4b are further formed of the same component, it joins firmly. Consequently, the interlayer 4 who changes from 1st interlayer 4a and the 2nd interlayer 4b to the ceramic base material 2 is minded. it becomes possible to join the anticorrosion material 3 very firmly, and it prevents effectively that the anticorrosion material 3 exfoliates from the ceramic base material 2 -- having -- corrosion resistance -- a member 1 can be used for stability over a long period of time

[0035] Moreover, although the aforementioned interlayer 4 was formed in two layers, 1st interlayer 4a and 2nd interlayer 4b, it is not limited to such two-layer structure, and the interlayer of two or more layers may be made to intervene between 1st interlayer 4a and 2nd interlayer 4b. In this case, if it considers so that the content of the ceramic base-material component of a layer and anticorrosion material component which are made to intervene may contain a ceramic base-material component in the layer near the ceramic base material 2 and many anticorrosion material components may be made to contain in the layer near anticorrosion material, it will become possible to join firmly between each class which forms the ceramic base material 2, the 1st interlayer 4a and the anticorrosion material 3, the 2nd interlayer 4b, and an interlayer 4. An anticorrosion material component 95 volume % content for a ceramic base-material component to 1st interlayer 4a at 95 volume % and 2nd interlayer 4b For example, when it carried out and forms, this -- the side which touches 1st interlayer 4a between 1st interlayer 4a and 2nd interlayer 4b -- a ceramic base-material component -- 65 volume % -- It becomes possible to join between each interlayer still more firmly by allotting the interlayer who did 65 volume % content of 35 volume % and an anticorrosion material component for the ceramic base-material component to the side which touches 2nd interlayer 4b again in the interlayer who did 35 volume % content of an anticorrosion material component. [0036] next, the above-mentioned corrosion resistance -- the concrete process of a member 1 is explained [0037] First, the raw material powder which serves as the ceramic base material 2, the anticorrosion material 3, and an interlayer 4 is adjusted.

[0038] (Adjustment of the raw material for ceramic base materials) Addition mixture of the organic binder of requests to the raw material powder which made the aluminum oxide as a principal component contain the oxidization silicon as a sintering acid, a magnesium oxide, a calcium oxide, etc. 0.1 to 10% of the weight, such as paraffin wax, a wax emulsion (wax + emulsifier), and PVA (polyvinyl alcohol), PEG (polyethylene glycol), is carried out, it corns by spray dry, and a raw material is adjusted.

[0039] (Adjustment of the raw material for anticorrosion material) After mixing aluminum oxide dust and yttria powder at a rate of a lower formula and carrying out temporary quenching at 1000-1600 degrees C, these are ground and 0.1-1.2 micrometers of mean particle diameters and the YAG powder of 2-5m²/g of BET specific surface areas] g are manufactured.

[0040] $A+B=10.365 < A \leq 0.3850.615 < B \leq 0.635A$: -- molar quantity B: of a yttria -- addition mixture of the molar quantity of an aluminum oxide, next the organic binder of requests to the aforementioned YAG powder, such as paraffin wax, PVA (polyvinyl alcohol), a wax emulsion (wax + emulsifier), and PEG (polyethylene glycol), is carried out, it corns by spray dry, and a raw material is adjusted

[0041] (Adjustment of the raw material for interlayers) A request of the above-mentioned raw material for ceramic base materials and the raw material for anticorrosion material carries out volume % [every] addition mixture, and the raw material for interlayers is adjusted.

[0042] In addition, in forming an interlayer 4 by the monostromatic, carry out 50 volume % [every] addition mixture of the raw material for ceramic base materials, and the raw material for anticorrosion material, and it adjusts the raw material for interlayers. moreover, in forming by two-layer [of 1st interlayer 4a and 2nd interlayer 4b] The raw material for interlayers which did [the raw material for interlayers which did 5-45 volume % mixture of 55 to 95 volume % and the raw material for anticorrosion material for the raw material for ceramic base materials as an object for 1st interlayer 4a] 5-45 volume % mixture of 55 to 95 volume % and the raw material for ceramic base materials for the raw material for anticorrosion material as an object for 2nd interlayer 4b is adjusted.

[0043] Next, each above-mentioned raw material is fabricated in a predetermined configuration for example, by die-press fabrication. The fabrication by the aforementioned die press presses this by the fixed pressure while being first filled up with the raw material for ceramic base materials in a die-press making machine. Form a ceramic base-material Plastic solid, and while being filled up with the raw material for

interlayers on the aforementioned ceramic base-material Plastic solid next, press this by the fixed pressure and an interlayer Plastic solid is formed on a ceramic base-material Plastic solid. Next, while being filled up with the raw material for anticorrosion material on the aforementioned interlayer Plastic solid, this is pressed by the fixed pressure, an anticorrosion material Plastic solid is formed on an interlayer Plastic solid, and the composite molding object which consists of a ceramic base-material Plastic solid, an interlayer Plastic solid, and an anticorrosion material Plastic solid by this is acquired.

[0044] and the corrosion resistance as a product which joined the anticorrosion material 3 through the interlayer 4 on the ceramic base material 2 when degreasing at 300-600 degrees C if needed and calcinating the aforementioned composite molding object at about 1500-1750 degrees C in air atmosphere after an appropriate time finally -- a member 1 is completed

[0045] In this case, if the thickness of the aforementioned anticorrosion material 3 can be made [200 micrometers or more] thick and thickness of this anticorrosion material 3 is set to 200 micrometers or more by adjusting the fill of the raw material for the anticorrosion material 3, it will become possible [presenting prolonged use from consumption of anticorrosion material taking time].

[0046] in addition -- if, as for the aforementioned anticorrosion material 3, the thickness is set to less than 200 micrometers -- an interlayer 4 top -- the anticorrosion material 3 -- uniform thickness -- and an interlayer's 4 front face -- perfect -- a wrap -- it is difficult to form like -- becoming -- corrosion resistance - there is a danger that the corrosion resistance of a member 1 will deteriorate. Moreover, if the thickness of the anticorrosion material 3 exceeds 30mm, in case the anticorrosion material 3 which calcinates the raw material for anticorrosion material and consists of a sintered compact will be formed, there is a danger that it will become difficult to degrease completely the organic binder contained in the raw material for anticorrosion material, and it will weaken the mechanical strength of the anticorrosion material 3.

Therefore, as for the aforementioned anticorrosion material 3, what the thickness is set to 200 micrometers or more for is desirable, and it is good to consider as the range of 200 micrometers - 30mm more suitably.

[0047] Moreover, if the thickness is set to 200 micrometers or more, the aforementioned interlayer 4 becomes possible [forming the interlayer 4 of uniform thickness the whole region between the ceramic base material 2 and the anticorrosion material 3], and can join very firmly the ceramic base material 2 and the anticorrosion material 3 through an interlayer 4 by this. Therefore, as for the aforementioned interlayer 4, what the thickness is set to 200 micrometers or more for is desirable.

[0048] furthermore, the thing for which the forming method by the die press is adopted by the above-mentioned method -- corrosion resistance -- although the member 1 was manufactured, it is not limited to this and the forming methods, such as casting and injection molding, may be adopted

[0049] this corrosion resistance -- a member 1 -- wall material (chamber), such as semiconductor fabrication machines and equipment, -- It is used for an etching system as used suitable for a microwave introduction aperture, a focal ring, etc., for example, shown in drawing 3. Pour in a halogen system corrosive gas into a chamber 5, carry out the seal of approval of the RF power to the induction coil 9 currently wound around the surroundings, and gas is plasma-ized. RF power is given also like the lower electrode 7, bias is generated, plasma is brought together in about eight wafer in the focal ring 6, and desired etching processing is performed. In order that the plasma generated with this equipment may contact a chamber 5 and the focal ring 6, especially these parts tend to receive corrosion. then, a chamber 5 and the focal ring 6 -- the aforementioned corrosion resistance -- the corrosion resistance which was excellent by forming by the member 1 can be provided, and it can prevent that a chip, a crack, etc. arise at the time of installation and washing

[0050]

[Example] Next, the example of this invention is explained.

(Example 1)

(Raw material for ceramic base materials) Oxidization silicon (SiO_2), a magnesium oxide (MgO), and a calcium oxide (CaO) are contained as a sintering acid, addition mixture of polyvinyl alcohol (PVA), a polyethylene glycol (PEG), and the wax emulsion (wax + emulsifier) is carried out as an organic binder, respectively, it comes to the aluminum oxide whose purity a mean particle diameter is 1-15 micrometers, and is 95 - 99 % of the weight as a principal component by spray dry, and a raw material is adjusted to it.

[0051] (Raw material for anticorrosion material) A cerium oxide (CeO) is added as a sintering acid to a kind of YAG, a yttria (Y_2O_3), a magnesia (MgO), and a spinel (MgOAl_2O_3), addition mixture of PVA, PEG, and the wax emulsion is carried out as an organic binder, it comes by spray dry, and each raw material is adjusted.

[0052] (Raw material for interlayers) 50 volume % [every] addition mixture of the aforementioned raw

material for ceramic base materials and the raw material for anticorrosion material is carried out, respectively, and a raw material is adjusted.

[0053] Next, the raw material for ceramic base materials, the raw material for interlayers, and the raw material for anticorrosion material are fabricated by die-press fabrication one by one, and the composite molding object with which each consists of the diameter of 30mm, a ceramic base-material Plastic solid with a thickness of 5mm, an interlayer Plastic solid, and an anticorrosion material Plastic solid is acquired.

[0054] next, each corrosion resistance which consists of the component which calcinates at 1500-1750 degrees C for 5 hours, and a ceramic base material, an interlayer, and anticorrosion material show in Table 1 after degreasing the aforementioned composite molding object at 350 degrees C for 2 hours -- a member - the sample was manufactured

[0055] Moreover, the sample (sample No.9-12) which formed the anticorrosion material which makes YAG and a yttria a principal component, respectively by thermal spraying and CVD at 1mm in the diameter of 30mm and thickness on the ceramic base material (5mm in the diameter of 30mm, thickness) which consists of the nature sintered compact of an aluminum oxide as a conventional example was manufactured.

[0056] In addition, it asked for the relative density of the anticorrosion material of each sample by the following formula.

[0057]

$(\text{Sintered density/theoretical density}) \times 100 = \text{relative density (\%)}$

Furthermore, the coefficient of thermal expansion of the ceramic base material of each sample, an interlayer, and anticorrosion material was measured based on JISR1618 (measuring range is room temperature -1500 degree C).

[0058] And in order to evaluate the junction state of each sample, the heat cycle (room temperature -1400 degree C) was repeated and impressed to each sample, and that in which anticorrosion material exfoliated from the ceramic base material made O that in which impression of a heat cycle did not exfoliate that in which it exfoliated in xx and less than 20 - 30 times x and at least 60 times in less than 20 times.

[0059] Subsequently, in order to evaluate the corrosion resistance of each sample, while making the anticorrosion material of each sample into the mirror plane by lap processing, it set in RIE (Reactive Ion Etching) equipment and ** (ed) in plasma under Cl₂ gas atmosphere for 3 hours, and the etching rate per for 1 minute after the decrement of the weight of the anticorrosion material before and behind it was computed and evaluated.

[0060] The numeric value of the aforementioned etching rate evaluated by relative comparison when using the nature sintered compact of an aluminum oxide of 99.9 % of the weight of purity as an authentic sample, and setting the etching rate to 1. And or more for 0.8, x and 0.5 or less were made into O, and, as for the criteria of the evaluation, the etching rate made 0.3 or less O.

[0061] An evaluation result is shown in Table 1.

試料 No.	セラミック基材		中間層	耐食材			接合方法	接合状態 の評価	耐食材のエッ チングレート	耐食性 の評価
	主成分	熱膨張係数 (ppm/°C)	熱膨張係数 (ppm/°C)	主成分	熱膨張係数 (ppm/°C)	相対密度 (%)			(A/min)	
1	アルミナ	7.4	7.9	YAG	8.8	98	一体焼成	◎	0.24	◎
2	アルミナ	7.4	8.0	イットリア	8.7	98	一体焼成	◎	0.21	◎
3	アルミナ	7.4	8.3	YAG	9.3	99	一体焼成	◎	0.19	◎
4	アルミナ	7.4	8.4	イットリア	9.4	99	一体焼成	◎	0.13	◎
5	アルミナ	7.4	8.4	マグネシア	9.4	98	一体焼成	◎	0.50	○
6	アルミナ	7.4	8.3	マグネシア	9.5	98	一体焼成	◎	0.43	○
7	アルミナ	7.4	8.0	スピネル	8.8	98	一体焼成	◎	0.48	○
8	アルミナ	7.4	7.9	スピネル	8.4	99	一体焼成	◎	0.42	○
* 9	アルミナ	7.4	—	YAG	8.2	93	溶剤	××	0.96	×
* 10	アルミナ	7.4	—	イットリア	8.1	93	溶剤	××	0.92	×
* 11	アルミナ	7.4	—	YAG	8.2	95	CVD	×	0.87	×
* 12	アルミナ	7.4	—	イットリア	8.1	95	CVD	×	0.81	×
*耐食材の比較例 純度99.9重量%アルミナ									1.00	×

*印の試料は本発明の請求の範囲外である。

alumina

Spinel

MgO

Thermal
spray

Sintered body

[0062]

Though a heat cycle is repeatedly impressed in the case of this invention article (sample No.1-8) which prepared the interlayer between a ceramic base material and anticorrosion material since junction to a ceramic base material and anticorrosion material is very strong so that clearly from Table 1, anticorrosion material does not exfoliate from a ceramic base material, and prolonged use can be presented.

[0064] On the other hand, it turns out that anticorrosion material exfoliates from a ceramic base material in less than 30 times, and impression of a heat cycle cannot present prolonged use with the conventional example (sample No.9-12) which made anticorrosion material put on a ceramic base material by thermal spraying or CVD.

[0065] Moreover, the relative density of anticorrosion material is 98% or more, and an etching rate is below 0.5 (A/min), and since this invention article (sample No.1-8) is extremely excellent in corrosion resistance, consumption of anticorrosion material can present few prolonged use with it.

[0066] On the other hand, conventionally, an etching rate will be inferior to corrosion resistance above 0.81 (A/min), and will exhaust elegance (sample No.9-12) for a short period of time.

[0067] (Example 2)

(Raw material for ceramic base materials) Oxidization silicon (SiO₂), a magnesium oxide (MgO), and a calcium oxide (CaO) are contained as a sintering acid, addition mixture of polyvinyl alcohol (PVA), a polyethylene glycol (PEG), and the wax emulsion is carried out as an organic binder, respectively, it comes to the aluminum oxide whose purity a mean particle diameter is 1-15 micrometers, and is 95 - 99 % of the weight as a principal component by spray dry, and a raw material is adjusted to it.

[0068] (Raw material for anticorrosion material) A cerium oxide (CeO) is added as a sintering acid to a

kind of YAG (yttrium aluminum garnet), a yttria (Y_2O_3), and a spinel ($MgO \cdot Al_2O_3$), addition mixture of PVA, PEG, and the wax emulsion is carried out as an organic binder, it comes by spray dry, and each raw material is adjusted.

[0069] (Raw material for interlayers) Addition mixture is carried out at a rate which shows the aforementioned raw material for ceramic base materials, and the raw material for anticorrosion material in Table 2, and the raw material for interlayers which forms the 1st interlayer and the 2nd interlayer is adjusted.

[0070] Next, the raw material for ceramic base materials, the raw material for interlayers, and the raw material for anticorrosion material are fabricated by die-press fabrication one by one, and the composite molding object with which each consists of the diameter of 30mm, a ceramic base-material Plastic solid with a thickness of 2mm, an interlayer Plastic solid, and an anticorrosion material Plastic solid is acquired.

[0071] next, each corrosion resistance which consists of the component which calcinates at 1500-1750 degrees C for 5 hours, and a ceramic base material, an interlayer, and anticorrosion material show in Table 2 after degreasing the aforementioned composite molding object at 350 degrees C for 2 hours -- a member - the sample was manufactured

[0072] In addition, it asked for the relative density of the anticorrosion material of each sample by the following formula.

[0073]

(Sintered density/theoretical density) × 100 = relative density (%)

Furthermore, the coefficient of thermal expansion of the ceramic base material of each sample, an interlayer, and anticorrosion material was measured based on JISR1618 (measuring range is room temperature -1500 degree C).

[0074] And in order to evaluate the junction state of each sample, the heat cycle (room temperature -1400 degree C) was repeated and impressed to each sample, and that in which anticorrosion material exfoliated from the ceramic base material made O that in which impression of a heat cycle did not exfoliate that in which that in which it exfoliated in xx and less than 20 - 30 times was exfoliated in x and 60 - less than 90 times O and at least 90 times in less than 20 times.

[0075] Subsequently, in order to evaluate the corrosion resistance of each sample, while making the anticorrosion material of each sample into the mirror plane by lap processing, it set in RIE (Reactive Ion Etching) equipment and etched in plasma under Cl₂ gas atmosphere for 3 hours, and the etching rate per for 1 minute after the decrement of the weight of the anticorrosion material before and behind it was computed.

[0076] The numeric value of the aforementioned etching rate showed the value which carried out relative comparison when using the nature sintered compact of an aluminum oxide of 99.9 % of the weight of purity as an authentic sample, and setting the etching rate to 1.

[0077] An evaluation result is shown in Table 2.

試料 No.	セラミックス基板		第1の中間層		第2の中間層		被食材			接合状態 の評価	被食材の エッチング レート (Å/min)
	主成分	熱膨張係数 (ppm/°C)	セラミックス 成分 (ppm/°C)	熱膨張係数 (ppm/°C)	被食材 成分 (ppm/°C)	熱膨張係数 (ppm/°C)	主成分	熱膨張係数 (ppm/°C)	相対 密度 (%)		
21	7417	7.4	セラミックス成分及び被食材成分をそれぞれ 50体積%ずつ含有した1層から成る中間層 熱膨張係数 8.3 (ppm/°C)	5.9	被食材成分 成分 (ppm/°C)	8.3	YAG	9.3	98	○	0.24
22	7417	7.4	セラミックス成分及び被食材成分をそれぞれ 50体積%ずつ含有した1層から成る中間層 熱膨張係数 8.1 (ppm/°C)	5.9	被食材成分 成分 (ppm/°C)	8.3	YAG	9.3	98	○	0.34
23	7417	7.4	7.5	7.7	5.9	8.3	YAG	9.3	98	○	0.24
24	7417	7.4	8.6	8.3	5.9	9.2	YAG	9.3	98	◎	0.24
25	7417	7.4	5.6	8.3	7.5	8.8	YAG	9.3	98	◎	0.24
26	7417	7.4	4.6	8.4	9.5	9.2	YAG	9.3	98	○	0.24
27	7417	7.4	5.6	8.3	7.5	8.8	YAG	9.3	98	○	0.24
28	7417	7.4	7.5	7.8	5.9	8.4	YAG	9.3	98	◎	0.24
29	7417	7.4	9.7	7.4	7.4	8.8	YAG	9.3	98	○	0.24
30	7417	7.4	1.55	8.3	9.5	9.2	YAG	9.3	98	◎	0.24
31	7417	7.4	1.55	5.3	7.5	8.8	YAG	9.3	98	◎	0.24
32	7417	7.4	8.6	7.5	5.5	8.4	YAG	9.3	98	◎	0.24
33	7417	7.4	5.6	8.0	5.5	8.2	YAG	9.3	98	◎	0.24
34	7417	7.4	7.5	7.7	7.5	8.6	YAG	9.3	98	◎	0.48
35	7417	7.4	7.5	7.7	5.5	8.2	YAG	9.3	98	◎	0.48
36	7417	7.4	9.5	7.5	9.5	8.3	YAG	9.3	98	◎	0.48
37	7417	7.4	9.7	7.4	6.6	8.3	YAG	9.3	98	◎	0.48
38	7417	7.4	9.6	7.5	8.0	7.8	YAG	9.3	98	◎	0.48
39	7417	7.4	1.55	8.1	9.5	5.5	YAG	9.3	98	◎	0.21
40	7417	7.4	9.5	7.5	5.5	8.2	YAG	9.3	98	◎	0.21

alumina

Y2O3

Spinel

[0079] A ceramic base-material component 55-95 volume % content between a ceramic base material and anticorrosion material so that clearly from Table 2 With the 1st interlayer who does An anticorrosion material component 55-95 volume % content what allotted the 2nd interlayer who does (39 sample No.24, 25, 27, 30-36, 40) A ceramic base material and anticorrosion material have joined very firmly, though a heat cycle is impressed repeatedly, anticorrosion material does not exfoliate from a ceramic base material, and prolonged use can be presented.

[0080]

[Effect of the invention] The ceramic base material which consists of the nature sintered compact of an aluminum oxide according to the corrosion-resistant member of this invention, the [periodic-table] -- the [2a group or] -- the anticorrosion material which consists of a sintered compact which makes a principal component at least one sort of 3a group element It consists of the sintered compact containing the aforementioned ceramic base-material component and the anticorrosion material component, junction nature is good to both ceramic base material and anticorrosion material, and since it joined through the interlayer who has a coefficient of thermal expansion among both, junction of the anticorrosion material to a ceramic base material is very strong. Therefore, though the heat at the time of use is impressed to a corrosion-resistant member, anticorrosion material does not exfoliate from a ceramic base material, and a corrosion-resistant member can be used, always being stabilized.

[0081] the corrosion resistance at the time of impression of heat when the coefficient-of-thermal-expansion difference of a ceramic base material, an interlayer and anticorrosion material, and an interlayer is especially carried out in 1 ppm/degree C or less, respectively -- the junction nature of the ceramic base material of a member and anticorrosion material can be made with a much more good thing, and it becomes possible to use a corrosion-resistant member for stability further

[0082] Moreover, the aforementioned interlayer is made into at least 2 layer structures of the 1st interlayer who touches a ceramic base material, and the 2nd interlayer who touches anticorrosion material. If a ceramic base-material component is done at the 1st interlayer of the above and 55 to 95 volume % and the 2nd interlayer are made to do 55-95 volume % content of an anticorrosion material component It becomes possible considering junction of anticorrosion material [as opposed to / can strengthen the bonding strength between the ceramic base material, between the 1st interlayer and anticorrosion material, between the 2nd interlayer and the 1st, and 2nd interlayer, and / a ceramic base material by this] as a very firm thing to use a corrosion-resistant member for stability further.

[0083] furthermore, the aforementioned anticorrosion material -- the [periodic-table] -- the [2a group or] -- since it is formed with the sintered compact which makes a principal component at least one sort of 3a group element, if relative density of anticorrosion material can be made into 98% or more and relative density of anticorrosion material is made into 98% or more, existence of an open pore will almost be lost and corrosion resistance will become what was extremely excellent

[0084] furthermore, the aforementioned anticorrosion material -- the [periodic-table] -- the [2a group or] -- since it is formed with the sintered compact which makes a principal component at least one sort of 3a group element, if the thickness of anticorrosion material can be formed in a thick thing 200 micrometers or more and thickness of anticorrosion material is set to 200 micrometers or more, it will become possible to present prolonged use from consumption of anticorrosion material taking time